

Claims

[c1] 1. A improved method for reduction and/or removal of permanganate-reducing compounds (PRC's), C ar-boxylic acids and CC₂₂₂ ayl iodide compounds formed in the carbonylation of a carbonylatable reactant selected from the group consisting of methanol, methyl acetate, methyl formate and dimethyl ether and mixtures thereof to an acetic acid product, the products of said carbonylation including a volatile phase that is distilled to yield a purified acetic acid product and a first overhead comprising methyl iodide, water and at least one PRC, wherein the improvement comprises the steps of:

- (a) distilling at least a portion of the first overhead to produce a second overhead stream comprising methyl iodide, dimethyl ether, and said at least one PRC;
- (b) extracting the second overhead stream with water to form a first raffinate and a first aqueous extract stream containing said at least one PRC; and
- (c) extracting the first raffinate with water to form a second raffinate and a second aqueous extract stream containing said at least one PRC.

[c2] 2. The method of claim 1, wherein said at least one PRC

comprises acetaldehyde.

- [c3] 3. The method of claim 2, wherein sufficient acetaldehyde is removed from said volatile phase to maintain in said purified product a concentration of propionic acid less than about 400 parts per million by weight.
- [c4] 4. The method of claim 2, wherein sufficient acetaldehyde is removed from said volatile phase to maintain in said purified product a concentration of propionic acid less than about 250 parts per million by weight.
- [c5] 5. The method of claim 1, wherein the improvement further comprises introducing at least a portion of the second raffinate directly or indirectly into the reaction medium.
- [c6] 6. The method of claim 1, wherein extraction steps (b) and (c) are performed in separate vessels.
- [c7] 7. The method of claim 1, wherein extraction steps (b) and (c) are performed in at least one packed-bed extractor.
- [c8] 8. The method of claim 1, wherein extraction steps (b) and (c) are performed on trays within a single extraction vessel.
- [c9] 9. The method of claim 1, wherein water for one of ex-

traction steps (b) and (c) comprises at least a portion of one of the aqueous extract streams.

- [c10] 10. The method of claim 1, further comprising at least one further step of extracting the second raffinate with water to provide a third aqueous extract and a third raffinate.
- [c11] 11. The method of claim 10, wherein water for the at least one further extraction step comprises at least a portion of one or more of said first, second and third aqueous extract streams.
- [c12] 12. The method of claim 1, wherein said first overhead comprises dimethyl ether.
- [c13] 13. The method of claim 1, further comprising the step of adding dimethyl ether to at least one stream associated with said distillation step (a).
- [c14] 14. The method of claim 1, wherein said distillation step (a) further comprises the step of forming dimethyl ether during the distillation.
- [c15] 15. The method of claim 1, wherein said distillation step (a) comprises at least two sequential distillation steps.
- [c16] 16. The method of claim 1, wherein said second overhead comprises an amount of dimethyl ether effective to

reduce the solubility of methyl iodide in at least one of said aqueous extract streams.

[c17] 17. A process for producing acetic acid, comprising the steps of:

- (a) carbonylating at least one reactant selected from the group consisting of methanol, methyl acetate, methyl formate and dimethyl ether in a reactor containing a suitable reaction medium comprising an organic iodide;
- (b) separating the products of said carbonylation into a volatile product phase comprising acetic acid, and a less volatile phase;
- (c) distilling said volatile product phase to yield a purified acetic acid product and a first overhead comprising said organic iodide and at least one permanganate reducing compound (PRC);
- (d) distilling at least a portion of the first overhead to produce a PRC-enriched second overhead, said second overhead further comprising dimethyl ether; and
- (e) extracting the second overhead with water, wherein step (e) comprises at least two consecutive extraction steps, each extraction step comprising contacting the second overhead with water and separating therefrom an aqueous stream comprising said at least one PRC.

- [c18] 18. The method of claim 17, wherein said at least one PRC comprises acetaldehyde.
- [c19] 19. The method of claim 18, wherein sufficient acetaldehyde is removed from said volatile phase to maintain in said purified product a concentration of propionic acid less than about 400 parts per million by weight.
- [c20] 20. The method of claim 18, wherein sufficient acetaldehyde is removed from said volatile phase to maintain in said purified product a concentration of propionic acid less than about 250 parts per million by weight.
- [c21] 21. The process of claim 17, further comprising recycling at least a portion of the extracted second overhead directly or indirectly to the reactor.
- [c22] 22. The process of claim 17, wherein the at least two extraction steps are performed in separate vessels.
- [c23] 23. The process of claim 17, wherein extraction steps (b) and (c) are performed on trays within a single extraction vessel.
- [c24] 24. The process of claim 17, wherein the at least two extraction steps are performed in at least one packed-bed extractor.
- [c25] 25. The process of claim 17, wherein water for at least

one of the at least two extraction steps comprises at least a portion of one of the aqueous extract streams.

- [c26] 26. The method of claim 17, wherein said second overhead comprises an amount of dimethyl ether effective to reduce the solubility of methyl iodide in at least one of said aqueous extract streams.
- [c27] 27. The process of claim 17, wherein said first overhead comprises dimethyl ether.
- [c28] 28. The process of claim 17, further comprising the step of adding dimethyl ether to at least one stream associated with said distillation step (d).
- [c29] 29. The process of claim 17, wherein said distillation step (d) further comprises the step of forming dimethyl ether during the distillation.
- [c30] 30. A process for separating a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one permanganate reducing compound (PRC), said process comprising the steps of:
 - (a) distilling the mixture to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched overhead stream comprising dimethyl ether; and
 - (b) extracting the PRC enriched overhead stream with

water,
wherein step (b) comprises at least two consecutive extraction steps, each extraction step comprising contacting the PRC enriched overhead stream with water and separating therefrom an aqueous stream comprising said at least one PRC.

- [c31] 31. The method of claim 30, wherein said PRC enriched overhead stream comprises an amount of dimethyl ether effective to reduce the solubility of methyl iodide in said aqueous extract streams.
- [c32] 32. The process of claim 30, further comprising the step of adding dimethyl ether to the PRC enriched overhead stream before extracting the PRC enriched overhead stream with water.
- [c33] 33. The process of claim 30, wherein said at least one PRC comprises acetaldehyde.
- [c34] 34. The method of claim 30, wherein said distillation step (a) further comprises the step of forming dimethyl ether during the distillation.
- [c35] 35. The process of claim 30, further comprising the step of providing said mixture by separating a liquid composition into a light phase and a heavy phase, said liquid composition comprising water, acetic acid, methyl io-

dide, methyl acetate, methanol, and said at least one PRC, wherein the light phase comprises said mixture and the heavy phase comprises methyl iodide.

- [c36] 36. The process of claim 30, wherein said at least one PRC comprises acetaldehyde.
- [c37] 37. The process of claim 30, further comprising the steps of:
 - performing a liquid-vapor phase separation on the effluent of a methanol carbonylation reactor to form a vapor phase and a liquid phase;
 - distilling the vapor phase to form a first overhead and a liquid product; and
 - condensing at least a portion of the first overhead to provide said liquid composition.
- [c38] 38. The process of claim 37, further comprising recycling at least a portion of the extracted PRC enriched overhead directly or indirectly to the reactor.
- [c39] 39. The process of claim 30, wherein said at least one PRC comprises acetaldehyde.
- [c40] 40. The method of claim 39, wherein sufficient acetaldehyde is removed from said volatile phase to maintain in said purified product a concentration of propionic acid less than about 400 parts per million by weight.

[c41] 41. The method of claim 39, wherein sufficient acetaldehyde is removed from said volatile phase to maintain in said purified product a concentration of propionic acid less than about 250 parts per million by weight.